Trace Metals Micro-Heterogeneous Distribution in Rod Wax Deposits Formed in an Oil-Producing Well, Anadarko Basin, Oklahoma – A Micro-Beam X-Ray Fluorescence (XRF) Study

E. Chouparova, H. Feng and K. Jones (BNL), and A. Lanzirotti (U. of Chicago)

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The focus of the present study is on characterization by synchrotron x-ray fluorescence spectroscopy (XRF) of trace metal micro-distributions in a series of rod wax deposits formed at different depths (temperatures) in the same oil-producing well. In the present study, the monochromatic beam at the X26A beamline was focused to a spot size with horizonal and vertical dimensions of 150 µm and 350 µm, respectively. Data were obtained by XRF spectral analysis on individual spots and regions of interest line scans. XRF spectra were fit using the Naval Research Laboratory software available at the X26A beamline (NRLXRF), as a result of which quantitative estimates of peak areas were obtained. Since the wax deposits analyzed represent very complex and heterogeneous mixtures, we performed extensive sensitivity analysis to evaluate the ranges of uncertainty in peak area estimation by varying parameters for density, composition, and thickness. The normalized peak areas, which are proportional to concentration, for each metal were used to construct depth profiles of the metal distributions in studied series of wax deposits. We attempted to answer and/or further clarify the following questions: What are the major metals in the deposit series? Is their distribution heterogeneous within the same sample and among the samples? What is the scale of heterogeneity? Is there partitioning of metals to organic and inorganic phases?

The results of the study show that within individual samples, metals are heterogeneously distributed, and partitioning of elements is observed on a scale of several hundred microns. A preferential enrichment with different metals is observed at different spots in the same sample. For example, (Sr, Ca, Zn, Ni, and Mn), (Fe, Mn, Ti, V, and Si), (Cu and Pb), and Pb are found to be preferentially concentrated at different points of the same sample (Figure 1). Increased abundances of Sr and Ca at specific points may reflect the presence of inorganic carbonate particles. The high abundance of Fe associated with Si and Ti at another point may reflect the presence of clay particles (silicates) and/or metal particles (e.g., corrosion products from the well). Depth profiles of metals distributions in the wax deposit series demonstrate significant differences between the deepest two deposits and the shallower ones. The relative abundance of metals in the shallower wax deposits decreases in the order Fe≈Pb>>Br≈Sr>>Cu≈Zn>Ni>Ir>Ca>Mn. The deepest two deposits are characterized with several orders of magnitude higher abundances of Fe, Ca, and Sr, and presence of V, Ti, and Cr, which are not detected in the shallower deposits. In addition, a significant variability in vanadium abundances among different spots of the same sample is observed. Two possible sources for the significantly higher abundances of Fe, Ca, and Sr in the deepest deposit could be attributed to: (a) an increased abundance of carbonate particles originating from the oil-producing reservoir (for higher Ca and Sr content), (b) an old iron bridge plug in the well below the producing Viola formation, set before the last re-completion in the well (for higher Fe content).

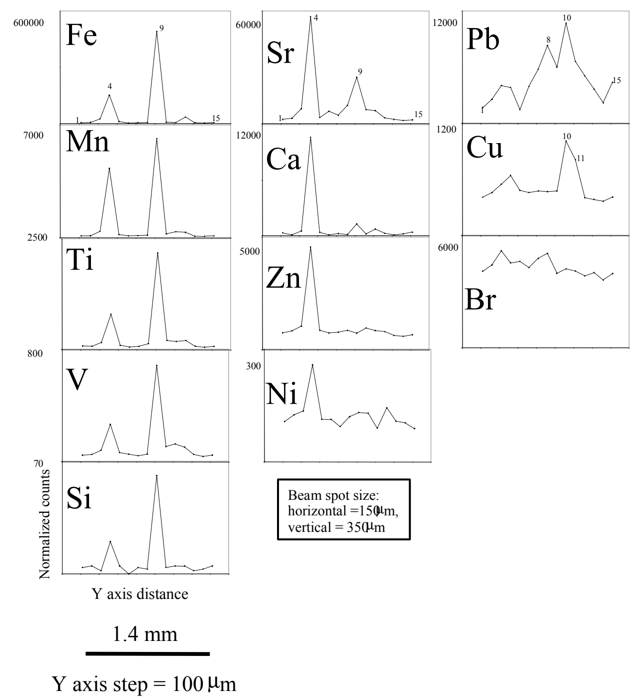

Figure 1. The line scan results demonstrate heterogeneity in metals distribution on a scale of several hundred microns within the same wax deposit sample. A preferential enrichment with different metals is observed at: point 4 – Sr, Ca, Zn, Ni and Mn; point 9 – Fe, Mn, Ti, V and Si; points 10 and 11 – Cu and Pb; point 8 – Pb.

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